

FATIGUE BEHAVIOUR IN DIFFERENT MOISTURE CONDITIONS OF A WOVEN HEMP FIBRE REINFORCED EPOXY COMPOSITE.

R. Barbière, F. Touchard, L. Chocinski-Arnault

Département Physique et Mécanique des Matériaux, Institut Pprime
CNRS-ENSMA-Université de Poitiers UPR 3346, ISAE-ENSMA
1 Avenue Clément Ader, 86961 Chasseneuil, France
e-mail : romain.barbiere@ensma.fr
e-mail : fabienne.touchard@ensma.fr
e-mail : laurence.chocinski@ensma.fr

Keywords: fatigue life, water uptake, eco-composite, hemp

Abstract

The purpose of this work is to characterise the influence of moisture on the fatigue behaviour of a woven hemp fibre reinforced epoxy composite. In a first time water uptake of this composite has been studied. Then two types of conditioning, before and during fatigue testing, have been studied for the $[\pm 45]_7$ composites; the so-called “non-aged samples” have been stored and then submitted to fatigue tests at ambient temperature and hygrometry, and the so-called “water-aged” samples have been tested in 95% of relative humidity atmosphere after water immersion until saturation. The fatigue behaviour is affected by the moisture conditions: for a given fatigue stress level, the fatigue lifetime is shorter for the “water-aged” samples. Fatigue damage mechanisms have been analysed by combining different techniques: acoustic emission monitoring (AE), post-mortem scanning electron microscopy (SEM) and X-ray micro-tomography. Although both types of samples show similar fracture surfaces, there are more cracks in the “water-aged” samples. Moreover the appearance and evolution of damage during fatigue test, deduced from AE analysis, are different: for the “water-aged” specimens damage appears from the beginning of the fatigue test and develops rapidly whereas for the “non-aged” ones the damage occurrence is delayed and progresses more slowly.

1. Introduction

Natural fibres, such as plant fibres, have emerged as a lightweight, low cost, and comparable property-wise alternative to glass fibres in composites [1]. However, a crucial issue with plant fibre composites is their durability when they are subjected to moisture; the plant fibres are hydrophilic and the matrix is comparatively hydrophobic. This difference of properties induces a decrease in the interfacial characteristics compared to glass fibre composites. The sensitivity of plant fibre composites to moisture leads to changes in their mechanical properties [2-4], and the fatigue behaviour of these composites has yet to be studied.

The objective of this work is to study the fatigue behaviour of a woven hemp/epoxy composite under two conditionings; some samples are water-aged by immersion in water and tested in a humid environment, and others are kept in ambient atmosphere and tested at ambient hygrometry (non-aged samples).

2. Materials and techniques

The composite of this study is constituted of seven plies of plain woven hemp fabric impregnated with epoxy resin. The hemp fabric has a fabric weight of $290 \pm 10 \text{ g/m}^2$. The epoxy resin is an EPOLAM 2020 from Axson Technologies with density of 1.10 g/cm^3 . The hemp fabric is pre-dried at 40°C for 24 h and the composite plates are manufactured by vacuum infusion. The fibre volume fraction is $40 \pm 8\%$.

Results are presented for a composite plate made with a stacking sequence of $[\pm 45]_7$. This plate has the warp direction of each ply oriented alternately at $+45^\circ$ and -45° from the tensile axis (X axis). From these plates, rectangular specimens were cut with the dimensions of $140 \text{ mm} \times 20 \text{ mm} \times 3,6 \text{ mm}$. The jaws of test machine take 30 mm of each extremity.

Each water-aged sample is immersed in a bottle of water and hold by a nylon thread. Non-aged samples are conditioned at ambient temperature and humidity (respectively about 20°C and $48 \pm 5\% \text{ RH}$).

Fatigue tests were performed with an Instron 8802 equipped with a climatic chamber, to allow the control of temperature and hygrometry. Constant amplitude loads were applied in a sinusoidal waveform at the frequency of 1 Hz in order to limit selfgenerated heating in the specimen [5]. The stress ratio (R), i.e. ratio between minimum (σ_{\min}) and maximum (σ_{\max}) stresses, was equal to 0.01 for all tests. In order to obtain detailed analysis of the damage process, tests were instrumented with Acoustic Emission (AE) monitoring. This monitoring was performed by using an acquisition system of Physical Acoustics SA.

Post-mortem, fracture surface observations were performed on a JEOL 7000F field emission gun scanning electron microscope (FEG-SEM). Specimens were coated with gold/palladium by vacuum metallisation. Moreover, some specimens have been analysed by X-ray micro-tomography. Image acquisition was performed at Pprime Institute using an UltraTom CT scanner manufactured by RX Solutions (France). A $13 \mu\text{m}$ resolution has been used in this work.

3. Water absorption

Water uptake was measured by weighing samples periodically at ambient temperature with a precision balance (sensitivity of 0.01 mg). Weight evolution of samples was studied during 350 days. Water uptake M_t was obtained from (Eq. 1) where w_t is the mass of the sample at time t and w_0 the initial mass.

$$M_t = \frac{w_t - w_0}{w_0} \times 100 \quad (1)$$

Measurements showed good reproducibility. For example, results obtained for a $[\pm 45]_7$ sample are plotted in the figure 1. Results show that weight stabilization during water ageing occurs after 90 days. Therefore, water-aged samples have been immersed in water more than 90 days in order to reach saturation.

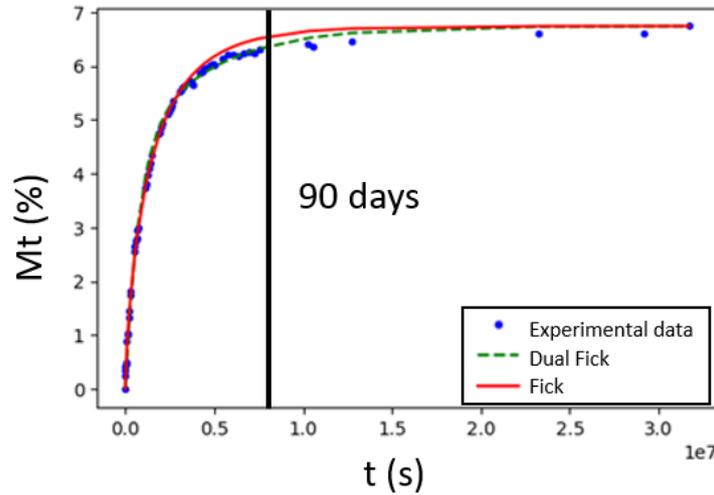


Figure 1. Experimental water uptake versus time of a $[\pm 45]_7$ woven hemp/epoxy composite immersed in water, with Fick and dual-Fick models.

The water uptake of the matrix can be described by a Fick law (Eq. 2, 3) [6], but the water uptake of the composite is better described by a dual-Fick law (Fig. 1). This law is the sum of two Fick laws with different diffusion coefficients and fractions of saturated mass uptake (Eq. 4) [7].

$$\frac{M_t}{M_\infty} = 1 - e^{\left(-7.3\left(\frac{Dt}{h^2}\right)^{0.75}\right)} \quad (2)$$

$$D = \pi\left(\frac{hk}{4M_\infty}\right)^2 \quad (3)$$

where M_∞ is the water uptake at saturation, M_t is the water uptake at time t , D is the coefficient diffusion, k is the initial slope of the M_t versus \sqrt{t} experimental curve and h is the thickness of the specimen.

$$M_t = \left(1 - \frac{8}{\pi^2} \sum_{n=0}^{\infty} \frac{1}{(2n+1)^2} \exp\left(\frac{-D_1(2n+1)^2\pi^2 t}{4h^2}\right)\right) \times M_{1\infty} + \left(1 - \frac{8}{\pi^2} \sum_{n=0}^{\infty} \frac{1}{(2n+1)^2} \exp\left(\frac{-D_2(2n+1)^2\pi^2 t}{4h^2}\right)\right) \times M_{2\infty} \quad (4)$$

with:

$$M_{1\infty} + M_{2\infty} = M_\infty \quad (5)$$

where D_1 and D_2 are the first and second diffusion coefficients respectively, M_t the water uptake at any time t , $M_{1\infty}$ and $M_{2\infty}$ are the fractions of saturated mass uptake M_∞ (Eq. 5).

4. Fatigue life

For each conditioning, the three applied levels of maximum stress (σ_{max}) have been determined from static tensile tests: 60%, 75% and 90% of the tensile strength σ_u for the $[\pm 45]_7$ samples. All fatigue tests were performed in the climatic chamber. The non-aged samples were tested at $21 \pm 1^\circ\text{C}$ and ambient hygrometry whereas, for the water-aged samples, fatigue tests were performed at $21 \pm 1^\circ\text{C}$ and 95% of relative humidity.

The Wöhler curves obtained for $[\pm 45]_7$ non-aged and water-aged samples are shown in figure 2.

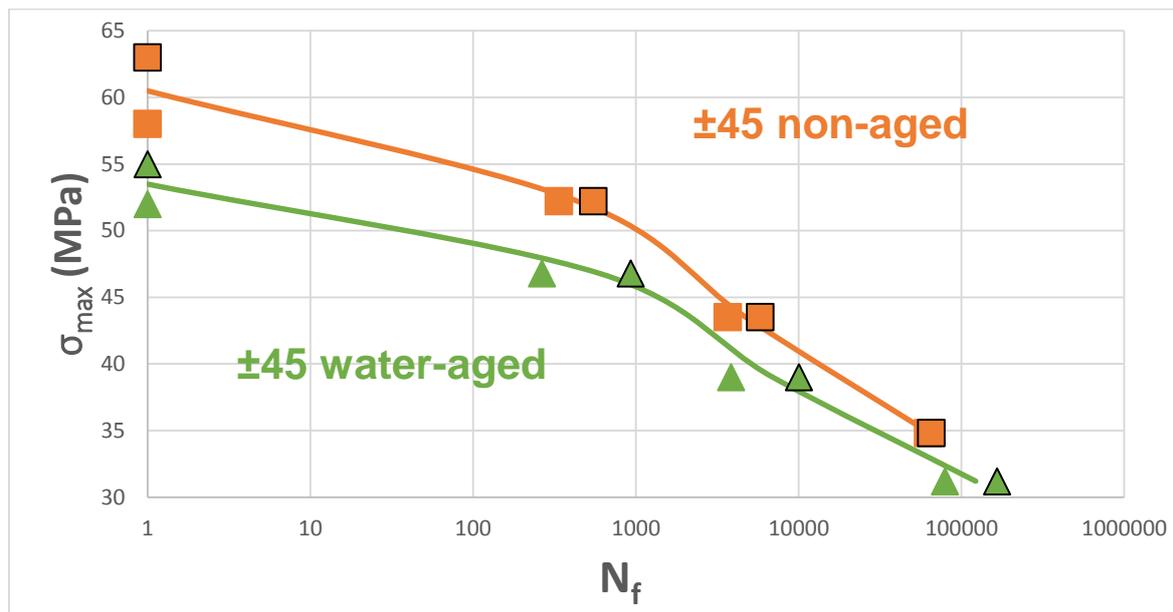


Figure 2. Wöhler curves of a $[\pm 45]_7$ woven hemp/epoxy composite for non-aged and water-aged specimens tested at ambient or at 95% of relative humidity respectively.

Figure 2 shows that the dispersion of fatigue results is not significant (less than a decade). Both non-aged and water-aged specimens show the same global fatigue behaviour. For all the stress levels, the higher the moisture content, the shorter the fatigue life. But this trend seems to decrease when it comes to lower stress levels. Indeed, from about 10^4 cycles, the non-aged and water-aged S-N curves tend to get closer.

5. Damage analysis

All the fatigue tests were monitored with acoustic emission AE. Figure 3 shows for example the cumulative number of acoustic events for the non-aged and aged $[\pm 45]_7$ samples tested at $0.9\sigma_u$. For comparison purpose, normalised values are presented. It can be seen that acoustic events appear earlier in the water-aged specimen and that the slope of the curve (i.e. the damage rate) is slightly higher (Fig. 3).

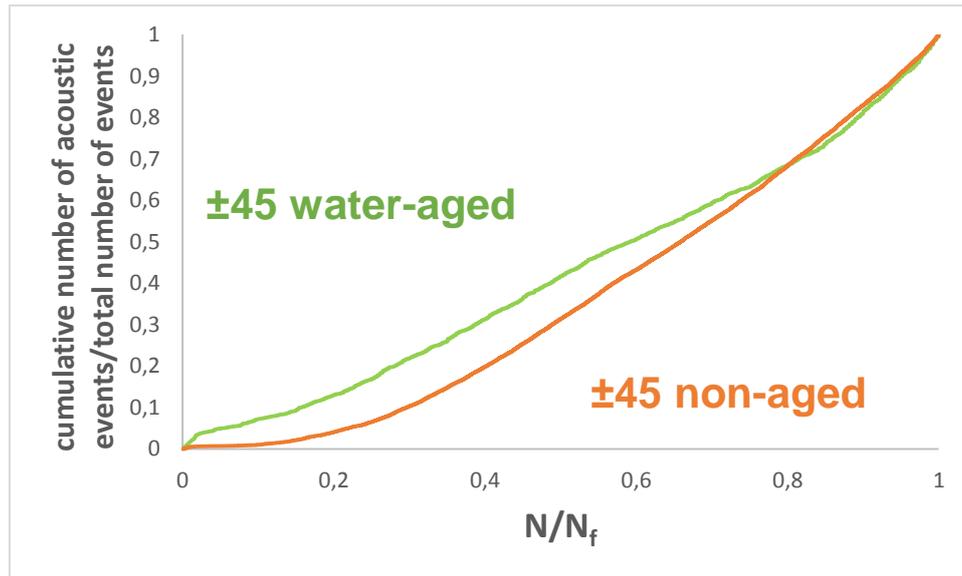


Figure 3. Cumulative number of acoustic events normalised by total events during fatigue tests for non-aged and water-aged $[\pm 45]_7$ woven hemp/epoxy composites.

Post-mortem SEM observations have been performed. Figure 4 presents two examples of fracture surfaces observed after fatigue test at $0.9\sigma_u$ for the $[\pm 45]_7$ composite. No significant difference between the non-aged and the aged samples can be detected from these SEM observations.

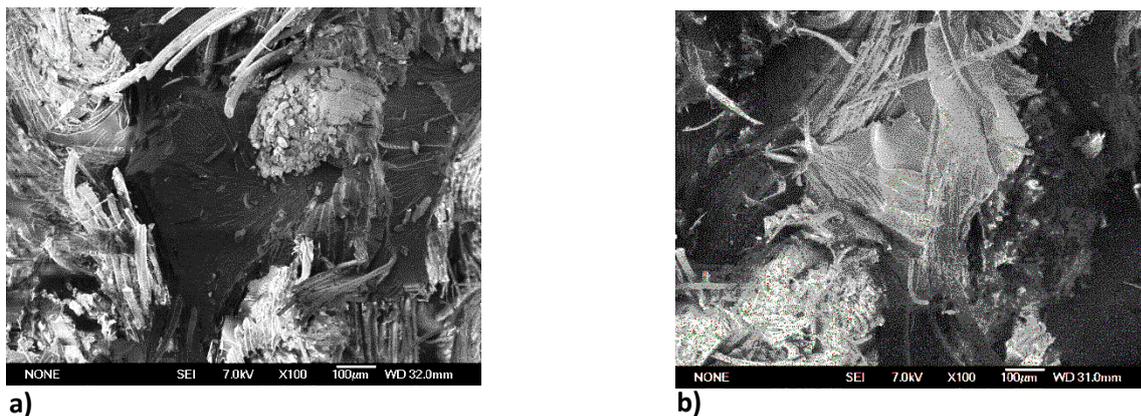


Figure 4. SEM observations of fracture surfaces after fatigue tests at $0.9\sigma_u$ of $[\pm 45]_7$ woven hemp/epoxy composites **a)** non-aged and tested at ambient hygrometry **b)** water-aged and tested at 95% of relative humidity.

An analysis by X-ray micro-tomography has also been performed, Micro-CT images after fatigue test at $0.9\sigma_u$ of non-aged and aged $[\pm 45]_7$ samples are presented in figure 5. It can be clearly seen that damage is far more developed in the sample that has been water-aged and tested at 95% of relative humidity. Many cracks, especially at the yarn/matrix interfaces, can be observed all along the free edges of the sample. Few cracks can also be observed in the core of the specimen. By contrast, only few damage on the free edges of the non-aged specimen can be seen. Moreover, the crack opening is far smaller in the non-aged specimen. These observations can explain the shorter fatigue life of the water-aged specimen compared with the non-aged specimen, as seen in figure 2.

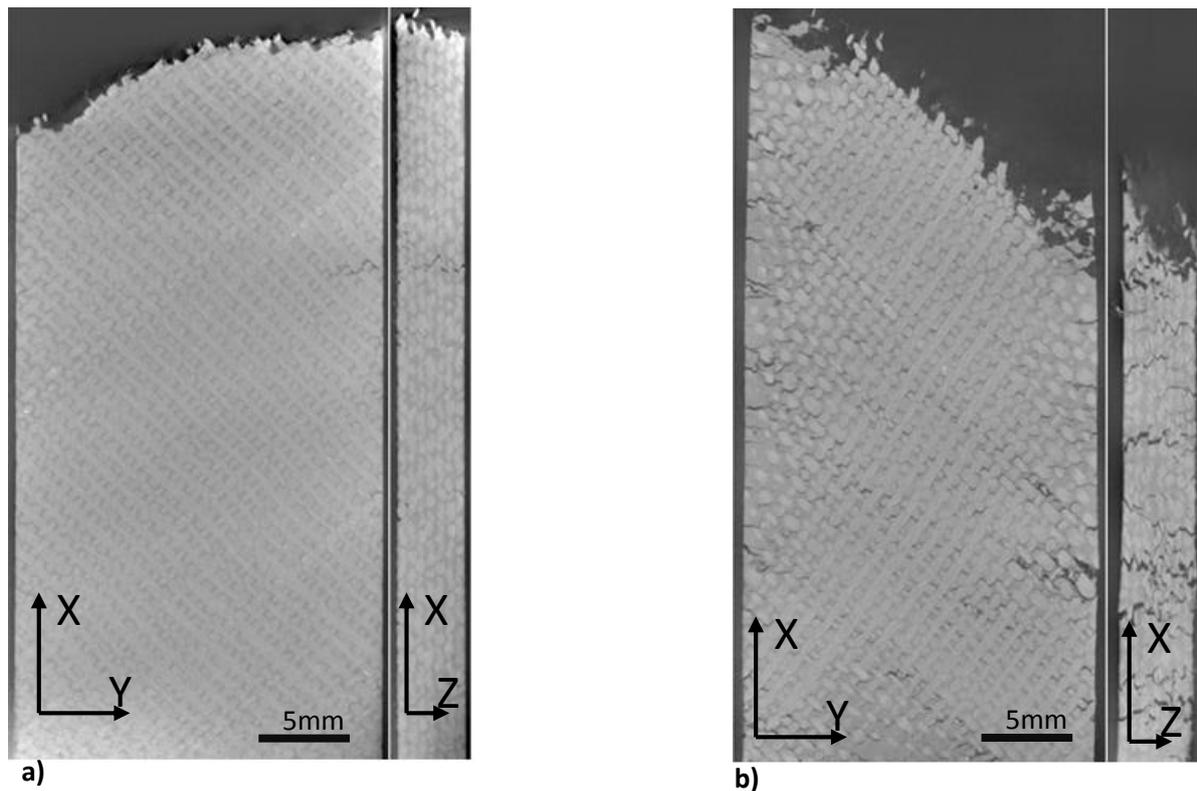


Figure 5. Micro-CT images after fatigue tests at $0.9\sigma_u$ of $[\pm 45]_7$ woven hemp/epoxy composites
a) non-aged and tested at ambient hygrometry b) water-aged and tested at 95% of relative humidity.

6. Conclusions

This study is focused on the influence of water absorption on tensile-tensile fatigue behaviour of woven hemp/epoxy composites. Results obtained on $[\pm 45]_7$ stacking sequence are presented. The gravimetric study has shown that the saturation is reached after 90 days in water, and that the mass uptake of the composite follows a dual-Fick law. The comparison of the Wöhler curves for a given applied stress highlights a longer fatigue life for the non-aged samples than for the water-aged ones. The AE results show that, contrary to the “non-aged” specimen behaviour, damage appears from the first fatigue cycles and develops rapidly in the “water-aged” composites. Post-mortem SEM observations of fracture surfaces depict no significant difference between the two types of specimens. However, micro-CT analysis reveals more numerous and open cracks in the water-aged composite than in the non-aged one.

References

- [1] P.Wambua, J.Ivens, and I.Verpoest. Natural fibres: can they replace glass in fibre reinforced plastics?. *Composites Science and Technology*, 63:1259-1264, 2003.
- [2] A.Shazad. Effects of Water Absorption on Mechanical Properties of Hemp Fiber Composites. *Polymer Composites*, 33:120-128, 2012.

- [3] M.Berges, R.Léger, V.Placet, V.Person, S.Corn, X.Gabrion, J.Rousseau, E.Ramasso, P.Ienny, and S.Fontaine. Influence of moisture uptake on the static, cyclic and dynamic behaviour of unidirectional flax fibre-reinforced epoxy laminate. *Composites part A*, 88:165-177, 2016.
- [4] A.Perrier, F.Touchard, L.Chocinski-Arnault, and D.Mellier, Quantitative analysis by micro-CT of damage during tensile test in a woven hemp/epoxy composite after water ageing, *Composites part A*, 102:118-127, 2017.
- [5] D.De Vasconcellos, F.Touchard, and L.Chocinski-Arnault. Tension–tension fatigue behaviour of woven hemp fibre reinforced epoxy composite: A multi-instrumented damage analysis. *International journal of fatigue*, 59:159-169, 2014.
- [6] C.H.Shen and G.S.Springer. Moisture Absorption and Desorption of Composite Materials. *J.Composite Materials*, 10:2-20, 1976.
- [7] A.Mubashar, I.A.Ashcroft, G.W.Critchlow, and A.D.Crocombe. Moisture absorption-desorption effects in adhesive joints. *International Journal of Adhesion & Adhesives*, 29:751-760, 2009.